

PATENT COOPERATION TREATY

PCT

NOTIFICATION OF ELECTION

(PCT Rule 61.2)

From the INTERNATIONAL BUREAU

To:

United States Patent and Trademark
Office
(Box PCT)
Crystal Plaza 2
Washington, DC 20231
ETATS-UNIS D'AMERIQUE

in its capacity as elected Office

Date of mailing (day/month/year) 01 December 1997 (01.12.97)	
International application No. PCT/GB97/01319	Applicant's or agent's file reference HL54557/001/CTV
International filing date (day/month/year) 14 May 1997 (14.05.97)	Priority date (day/month/year) 16 May 1996 (16.05.96)
Applicant HAYNS, Andrew, Bickford	

1. The designated Office is hereby notified of its election made:

☒ in the demand filed with the International Preliminary Examining Authority on:

05 November 1997 (05.11.97)

☐ in a notice effecting later election filed with the International Bureau on:2. The election ☒ was☐ was not

made before the expiration of 19 months from the priority date or, where Rule 32 applies, within the time limit under Rule 32.2(b).

The International Bureau of WIPO
34, chemin des Colombettes
1211 Geneva 20, Switzerland

Facsimile No.: (41-22) 740.14.35

Authorized officer

Aino Metcalfe

Telephone No.: (41-22) 338.83.38

PATENT COOPERATION TREATY 09/202500

PCT

INTERNATIONAL PRELIMINARY EXAMINATION REPORT

(PCT Article 36 and Rule 70)

Applicant's or agent's file reference HL54557/001/CTV	FOR FURTHER ACTION See Notification of Transmittal of International Preliminary Examination Report (PCT/IPEA/416)	
International application No. PCT/GB97/01319	International filing date (day/month/year) 14/05/1997	Priority date (day/month/year) 16/05/1996
International Patent Classification (IPC) or national classification and IPC B01D39/18		
Applicant AXHOLME RESOURCES LIMITED et al.		

1. This international preliminary examination report has been prepared by this International Preliminary Examining Authority and is transmitted to the applicant according to Article 36.



2. This REPORT consists of a total of 5 sheets, including this cover sheet.

- ☒ This report is also accompanied by ANNEXES, i.e., sheets of the description, claims and/or drawings which have been amended and are the basis for this report and/or sheets containing rectifications made before this Authority (see Rule 70.16 and Section 607 of the Administrative Instructions under the PCT).

These annexes consist of a total of 5 sheets.

3. This report contains indications relating to the following items:

- I ☒ Basis of the report
- II ☐ Priority
- III ☐ Non-establishment of opinion with regard to novelty, inventive step and industrial applicability
- IV ☐ Lack of unity of invention
- V ☒ Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement
- VI ☐ Certain documents cited
- VII ☐ Certain defects in the international application
- VIII ☐ Certain observations on the international application

Date of submission of the demand 05/11/1997	Date of completion of this report 05.02.98
Name and mailing address of the IPEA/  European Patent Office D-80298 Munich Tel. (+49-89) 2399-0, Tx: 523656 epmu d Fax: (+49-89) 2399-4465	Authorized officer Persichini, C Telephone No (+49-89) 2399-8617 

**INTERNATIONAL PRELIMINARY
EXAMINATION REPORT**

International application No. PCT/GB97/01319

I. Basis of the report

1. This report has been drawn on the basis of *(substitute sheets which have been furnished to the receiving Office in response to an invitation under Article 14 are referred to in this report as "originally filed" and are not annexed to the report since they do not contain amendments.)*:

Description, pages:

4-20	as originally filed		
1-3	as received on	20/07/1998 with letter of	17/07/1998

Claims, No.:

1-17	as received on	20/07/1998 with letter of	17/07/1998
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Drawings, sheets:

1-9	as originally filed
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2. The amendments have resulted in the cancellation of:

- ☐ the description, pages:
- ☐ the claims, Nos.:
- ☐ the drawings, sheets:

3. ☐ This report has been established as if (some of) the amendments had not been made, since they have been considered to go beyond the disclosure as filed (Rule 70.2(c)):

4. Additional observations, if necessary:

**INTERNATIONAL PRELIMINARY
EXAMINATION REPORT**

International application No. PCT/GB97/01319

V. Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement

1. Statement

Novelty (N)	Yes:	Claims	4-17
	No:	Claims	1-3
Inventive step (IS)	Yes:	Claims	
	No:	Claims	1-17
Industrial applicability (IA)	Yes:	Claims	1-17
	No:	Claims	

2. Citations and explanations

see separate sheet

**INTERNATIONAL PRELIMINARY
EXAMINATION REPORT - SEPARATE SHEET**

International application No. PCT/GB97/01319

1. DE-A-2 358 808 (D1) discloses a filter material (a material which is placed in a container and serves for filtering out contaminants from a fluid which is passed through the container is nothing else than a filter material; see D1, page 6, lines 9-13) comprising a matrix (Chambers English Dictionary: matrix = "that in which anything is embedded", eg a container) in which is dispersed a granular formulation of a material (according to eg claim 1 of D1 the modified cellulose mass is formed into small sized particles ["kleinteilig"]; it does not seem that there is a difference between "small sized particles" and a "granular formulation") comprising a base formed substantially of cellulose fibres onto which is adsorbed a composition comprising one or more aliphatic carboxylic acids having hydrocarbon chains consisting of 8 to 20 carbon atoms (see (D1), claim 1 and p.5, lines 1-5 and Example 16).

Claim 1 thus seems to lack novelty (Art.33(2) PCT).

However, even if a difference could be seen between the small sized particles of D1 and the "granular formulation" of claim 1, this difference would not be based on an inventive step under Art.33(3) PCT.

With regard to the handling and to the fluidic conditions (especially with regard to the pressure drop) the pelletization of filtering materials which are placed in a container and the filter activity of which is due to the adhesion of the components to be filtered (adsorption, absorption) to the surface of this material is usual. It is, therefore, evident for the skilled person to form pellets or granules from such a mass, if it has to be placed as a filtering fixed bed into a container.

2. The subject-matter of Independent method claim 10 differs from the teaching of (D1) on account only of the fact that the carboxylic acid is in powder form. In the absence, however, of any surprising effect (the advantages and disadvantages linked with the deposition of the carboxylic acid in dissolved form and in powder form, respectively, are well known in the art and consequently not surprising) attaching to this step, it does not represent an inventive advance over the process of (D1). Claim 11 thus lacks inventive step (Art.33(3) PCT). Analogous arguments apply to the subject-matter of independent method (use) claim 15.

3. With regard to document (D1) and the general knowledge of the man skilled in the art, the dependent claims do not appear to contain any features which, in combi-

**INTERNATIONAL PRELIMINARY
EXAMINATION REPORT - SEPARATE SHEET**

International application No. PCT/GB97/01319

nation with the features of any claim to which they refer, meet the requirements of the PCT in respect of novelty or inventive step.

INTERNATIONAL SEARCH REPORT

(PCT Article 18 and Rules 43 and 44)

Applicant's or agent's file reference HL54557/001/CTV	FOR FURTHER ACTION see Notification of Transmittal of International Search Report (Form PCT/ISA/220) as well as, where applicable, item 5 below.	
International application No. PCT/GB 97/ 01319	International filing date (day/month/year) 14/05/1997	(Earliest) Priority Date (day/month/year) 16/05/1996
Applicant AXHOLME RESOURCES LIMITED et al.		

This International Search Report has been prepared by this International Searching Authority and is transmitted to the applicant according to Article 18. A copy is being transmitted to the International Bureau.

This International Search Report consists of a total of 3 sheets.

☒ It is also accompanied by a copy of each prior art document cited in this report.

1. ☐ Certain claims were found unsearchable (see Box I).

2. ☐ Unity of invention is lacking (see Box II).

3. ☐ The international application contains disclosure of a nucleotide and/or amino acid sequence listing and the international search was carried out on the basis of the sequence listing

☐ filed with the international application.

☐ furnished by the applicant separately from the international application,

☐ but not accompanied by a statement to the effect that it did not include matter going beyond the disclosure in the international application as filed.

☐ Transcribed by this Authority

4. With regard to the title, ☒ the text is approved as submitted by the applicant.

☐ the text has been established by this Authority to read as follows:

5. With regard to the abstract,

☒ the text is approved as submitted by the applicant.

☐ the text has been established, according to Rule 38.2(b), by this Authority as it appears in Box III. The applicant may, within one month from the date of mailing of this International Search Report, submit comments to this Authority.

6. The figure of the drawings to be published with the abstract is:

Figure No. _____ ☐ as suggested by the applicant.

☐ because the applicant failed to suggest a figure.

☐ because this figure better characterizes the invention.

☒ None of the figures.

INTERNATIONAL SEARCH REPORT

International Application No
PCT/GB 97/01319

A. CLASSIFICATION OF SUBJECT MATTER

IPC 6 B01D39/18 B01D39/08 B01D25/26 B01J20/24

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC 6 B01D B01J C09K

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	DE 23 58 808 A (HOECHST AG) 5 June 1975 see page 1 - page 4; claim ---	1-3,11, 12,14,17
Y	US 4 018 679 A (BOLSING FRIEDRICH) 19 April 1977 see the whole document ---	1-3,11, 12,14,17
A	WO 91 08037 A (PURIFICATION PROD) 13 June 1991 see page 18 - page 19; claims 1-14 & EP 0 504 214 A cited in the application ---	1,4-7,13
A	US 3 647 084 A (MARTIN HENRY WOODS) 7 March 1972 see the whole document ---	8-10,18
	--- -/--	

☒ Further documents are listed in the continuation of box C.☒ Patent family members are listed in annex.

* Special categories of cited documents:

- "A" document defining the general state of the art which is not considered to be of particular relevance
- "E" earlier document but published on or after the international filing date
- "L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)
- "O" document referring to an oral disclosure, use, exhibition or other means
- "P" document published prior to the international filing date but later than the priority date claimed

"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.

"&" document member of the same patent family

Date of the actual completion of the international search

26 August 1997

Date of mailing of the international search report

08.09.97

Name and mailing address of the ISA

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Fax: (+ 31-70) 340-3016

Authorized officer

Cubas Alcaraz, J

INTERNATIONAL SEARCH REPORT

International Application No
PCT/GB 97/01319

C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	US 5 156 686 A (VAN SLYKE DONALD C) 20 October 1992 -----	1

INTERNATIONAL SEARCH REPORT

Information on patent family members

International Application No

PCT/GB 97/01319

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
DE 2358808 A	05-06-75	AT 335376 B AU 7554874 A BE 822567 A BR 7409850 A CH 602493 A FR 2252297 A JP 50084482 A NL 7414934 A SE 7414649 A ZA 7407503 A	10-03-77 20-05-76 26-05-75 25-05-76 31-07-78 20-06-75 08-07-75 28-05-75 27-05-75 28-01-76
US 4018679 A	19-04-77	DE 2328777 A DE 2328778 A AT 335375 B BE 815960 A CA 1041127 A CH 606380 A FR 2232517 A GB 1477209 A JP 1167626 C JP 50032075 A JP 58002000 B NL 7407581 A,B, SU 913934 A	13-02-75 23-01-75 10-03-77 30-09-74 24-10-78 31-10-78 03-01-75 22-06-77 08-09-83 28-03-75 13-01-83 10-12-74 15-03-82
WO 9108037 A	13-06-91	AT 112176 T CA 2068432 A DE 69013009 D DE 69013009 T EP 0504214 A ES 2065667 T GB 2238802 A,B JP 7010373 B JP 5503030 T KR 9612675 B US 5281437 A	15-10-94 07-06-91 03-11-94 02-02-95 23-09-92 16-02-95 12-06-91 08-02-95 27-05-93 24-09-96 25-01-94
US 3647084 A	07-03-72	CA 929111 A	26-06-73
US 5156686 A	20-10-92	US 5213625 A	25-05-93

Information on patent family members

PCT/GB 97/01319

01-06-93
10-08-93

INTERNATIONAL SEARCH REPORT

International Application No

PCT/GB 97/01319

A. CLASSIFICATION OF SUBJECT MATTER

IPC 6 B01D39/18 B01D39/08 B01D25/26 B01J20/24

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC 6 B01D B01J C09K

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

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AE Y	DE 23 58 808 A (HOECHST AG) 5 June 1975 see page 1 - page 4; claim ---	1-3,11, 12,14,17
AB Y	US 4 018 679 A (BOLSING FRIEDRICH) 19 April 1977 see the whole document ---	1-3,11, 12,14,17
AF A	WO 91 08037 A (PURIFICATION PROD) 13 June 1991 see page 18 - page 19; claims 1-14 & EP 0 504 214 A cited in the application ---	1,4-7,13
AC A	US 3 647 084 A (MARTIN HENRY WOODS) 7 March 1972 see the whole document ---	8-10,18
	--- -/--	

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- *A* document defining the general state of the art which is not considered to be of particular relevance
- *E* earlier document but published on or after the international filing date
- *L* document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)
- *O* document referring to an oral disclosure, use, exhibition or other means
- *P* document published prior to the international filing date but later than the priority date claimed

T later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

X document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

Y document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art

& document member of the same patent family

Date of the actual completion of the international search

26 August 1997

Date of mailing of the international search report

08.09.97

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Cubas Alcaraz, J

INTERNATIONAL SEARCH REPORT

International Application No
PCT/GB 97/01319

C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
<p>AA A</p>	<p>US 5 156 686 A (VAN SLYKE DONALD C) 20 October 1992</p> <p>-----</p>	<p>1</p>

INTERNATIONAL SEARCH REPORT

Information on patent family members

International Application No

PCT/GB 97/01319

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
DE 2358808 A	05-06-75	AT 335376 B	10-03-77
		→ AU 7554874 A	20-05-76
		BE 822567 A	26-05-75
		BR 7409850 A	25-05-76
		CH 602493 A	31-07-78
		FR 2252297 A	20-06-75
		JP 50084482 A	08-07-75
		NL 7414934 A	28-05-75
		SE 7414649 A	27-05-75
		→ ZA 7407503 A	28-01-76

US 4018679 A	19-04-77	DE 2328777 A	13-02-75
		DE 2328778 A	23-01-75
		AT 335375 B	10-03-77
		BE 815960 A	30-09-74
		CA 1041127 A	24-10-78
		CH 606380 A	31-10-78
		FR 2232517 A	03-01-75
		GB 1477209 A	22-06-77
		JP 1167626 C	08-09-83
		JP 50032075 A	28-03-75
		JP 58002000 B	13-01-83
		NL 7407581 A,B,	10-12-74
		SU 913934 A	15-03-82

WO 9108037 A	13-06-91	AT 112176 T	15-10-94
		CA 2068432 A	07-06-91
		DE 69013009 D	03-11-94
		DE 69013009 T	02-02-95
		EP 0504214 A	23-09-92
		ES 2065667 T	16-02-95
		GB 2238802 A,B	12-06-91
		JP 7010373 B	08-02-95
		JP 5503030 T	27-05-93
		KR 9612675 B	24-09-96
		US 5281437 A	25-01-94

US 3647084 A	07-03-72	CA 929111 A	26-06-73

US 5156686 A	20-10-92	US 5213625 A	25-05-93

Information on patent family members

PCT/GB 97/01319

Patent document
cited in search report

Publication date

Patent family member(s)

Publication date

US 5156686 A

US 5215596 A

01-06-93

US 5234577 A

10-08-93

Courtesy copy of the
International Preliminary
Examination Report with annexes
containing specification pages
1-3 and claims 1-17 to be used
in place of original pages 1-3
and the original claims for
examination in this case

-1-

LIQUID AND GAS PURIFICATION AND FILTRATION

5 The present invention relates to the removal of organic and other pollutants from liquids and gases, and in particular, but not exclusively, to the removal of such pollutants by a filtration system.

10 A number of strategies have been developed in the petrochemical industry for dealing with problems such as oil spillage and leakage, particularly at sea. Some methods, for example the use of detergents, simply aim to disperse the oil spillage as quickly as possible before too much damage has been done. It is, however, preferable to remove the oil from the water without
15 allowing it to disperse, since there are many toxic components in the oil which may cause harm to the environment. It is known to provide a granular material based on cellulose, which has oil-absorbing properties, the material being in a form suitable for sprinkling onto an oil spillage. Once the oil has been
20 absorbed, the material is gathered up and may be incinerated.

25 Oil spillages are not the only environmental problem faced by the petrochemical industry. There are many situations where it is desirable to remove components including organic pollutants (such as hydrocarbons) and heavy metal contaminants from produced water and water run-off before this water is released as effluent.

30 It is also desirable to remove such pollutants from liquids other than water and also from gases (e.g. air).

35 According to a first aspect of the present invention, there is provided a filter material comprising a matrix in which is dispersed a granular formulation of a material comprising a base formed

substantially of cellulose fibres onto which is adsorbed a composition comprising one or more aliphatic carboxylic acids having hydrocarbon chains consisting of 8 to 20 carbon atoms.

5 In preferred embodiments, the hydrocarbon chains of the one or more carboxylic acids consist of 10 to 18 carbon atoms. Particularly effective carboxylic acids have been found to include stearic acid $\text{CH}_3(\text{CH}_2)_{16}\text{COOH}$ and palmitic acid $\text{CH}_3(\text{CH}_2)_{14}\text{COOH}$.

10 According to a second aspect of the present invention, there is provided a method of producing a material comprising a base formed substantially of cellulose fibres onto which is adsorbed a composition comprising one or more aliphatic carboxylic acids
15 having hydrocarbon chains consisting of 8 to 20 carbon atoms, wherein one or more cellulosic materials are mixed together with a powdered formulation of the one or more carboxylic acids.

20 The granular material of the first aspect of the present invention may be formed by mixing together one or more cellulosic materials, for example virgin pulp and wood chips, together with the one or more carboxylic acids in powder form and, optionally, latex. The mixing is preferably undertaken in a hammer mill,
25 in which heat and friction assist the process whereby the carboxylic acid becomes adsorbed onto the cellulose fibres. It is thought that the carboxylic acids are adsorbed onto the surface of the cellulose fibres by way of the carboxyl $-\text{COOH}$ functional group, either
30 through hydrogen bonding or through the formation of cellulose esters containing an $-\text{O}-\text{CO}-\text{R}$ group formed with the hydroxyl $-\text{OH}$ groups on the cellulose rings. However the carboxylic acids are bonded to the cellulose fibres, the result is that the material of
35 the first aspect of the present invention comprises cellulose fibres from which project hydrophobic

hydrocarbon chains. When the material is applied to a mixture of water and hydrocarbon pollutants, the hydrophobic hydrocarbon tails of the carboxylic acid residues serve to attract the hydrocarbon pollutants to the material and to repel water, thereby providing the required separation. The material, incorporating the hydrocarbon pollutants, can then be gathered up and used as a fuelstock.

The matrix of the first aspect of the invention may be fabricated from a number of materials, including non-woven fibrous materials, open-cell foam materials or a cotton or viscose gauze. The unloaded matrix advantageously has a density not greater than 0.25gcm^{-3} , and preferably from 0.01 to 0.18gcm^{-3} . A particularly preferred matrix has a thickness of around 3mm and a density in the region of 0.1gcm^{-3} . The granular formulation of the material of the first aspect of the present invention may be incorporated into the matrix by bombardment across a pressure gradient as described in EP 0 504 214, the disclosure of which is hereby incorporated by reference into the present application. By incorporating the material of the first aspect of the present invention into a matrix to form a filter material, the available active surface area is increased so as to aid efficiency. Furthermore, dispersion of the material in the contaminated fluid is reduced because it is held within the matrix. In some embodiments, webs of the filter matrix are loaded to a density of around 1kgm^{-2} ; a density of $.925\text{kgm}^{-2}$ has been found to be particularly effective in certain circumstances. In other embodiments, a density of around 0.5kgm^{-2} has been found to be effective, particularly where the web of filter matrix has a thickness in the region of 3 or 4mm . Once

CLAIMS:

1. A filter material comprising a matrix in which is dispersed a granular formulation of a material comprising a base formed substantially of cellulose fibres onto which is adsorbed a composition comprising one or more aliphatic carboxylic acids having hydrocarbon chains consisting of 8 to 20 carbon atoms.

2. A material as claimed in claim 1, wherein the one or more aliphatic carboxylic acids have hydrocarbon chains consisting of 10 to 18 carbon atoms.

3. A material as claimed in claim 1 or 2, wherein the one or more carboxylic acids are selected from the group comprising stearic acid and palmitic acid.

4. A filter material as claimed in claim 1, 2 or 3, wherein the matrix comprises a non-woven fibrous material.

5. A filter material as claimed in claim 1, 2 or 3, wherein the matrix comprises an open-cell foam materials.

6. A filter material as claimed in claim 1, 2 or 3, wherein the matrix comprises a cotton or viscose gauze.

7. A filter column comprising a hollow core upon which is mounted an alternating stack of filter plates and discs of the filter material as claimed in any of claims 1 to 6, wherein the filter plates are adapted to allow passage of fluid from a circumferential region of the filter column to the hollow core by way of the discs of filter material.

8. A filter cartridge comprising a hollow core around which is wrapped one or more layers of a filter material as claimed in any one of claims 1 to 6.

9. A filter pod comprising a casing internally divided into two chambers by a carrier which supports at least one filter cartridge as claimed in claim 8,

the carrier and the at least one cartridge being arranged so that fluid can only pass from one chamber to the other by passing through both the hollow tubular core and the filter material of the at least one cartridge.

10. A method of producing a material comprising a base formed substantially of cellulose fibres onto which is adsorbed a composition comprising one or more aliphatic carboxylic acids having hydrocarbon chains consisting of 8 to 20 carbon atoms, wherein one or more cellulosic materials are mixed together with a powdered formulation of the one or more carboxylic acids.

11. A method according to claim 12, wherein the one or more cellulosic materials are selected from the group comprising wood chips and virgin pulp.

12. A method according to claim 10 or 11, wherein latex is added to the one or more cellulosic materials and the one or more carboxylic acids.

13. A method according to any one of claims 10 to 12, wherein mixing takes place in a hammer mill.

14. A method of cleaning a fluid by contacting the fluid with a material comprising a base formed substantially of cellulose fibres onto which is adsorbed a composition comprising one or more aliphatic carboxylic acids having hydrocarbon chains consisting of 8 to 20 carbon atoms, wherein one or more cellulosic materials are mixed together with a powdered formulation of the one or more carboxylic acids.

15. A method according to claim 14, wherein the fluid is air.

16. A method according to claim 15, wherein the fluid is water.

17. A filter cartridge comprising a container having a fluid input and a fluid output and including therebetween a quantity of the material of any one of claims 1 to 6.

Courtesy copy of the
International Application
as originally filed
with abstract

-1-

LIQUID AND GAS PURIFICATION AND FILTRATION

5 The present invention relates to the removal of organic and other pollutants from liquids and gases, and in particular, but not exclusively, to the removal of such pollutants by a filtration system.

10 A number of strategies have been developed in the petrochemical industry for dealing with problems such as oil spillage and leakage, particularly at sea. Some methods, for example the use of detergents, simply aim to disperse the oil spillage as quickly as possible before too much damage has been done. It is, however, preferable to remove the oil from the water without
15 allowing it to disperse, since there are many toxic components in the oil which may cause harm to the environment. It is known to provide a granular material based on cellulose, which has oil-absorbing properties, the material being in a form suitable for
20 sprinkling onto an oil spillage. Once the oil has been absorbed, the material is gathered up and may be incinerated.

25 Oil spillages are not the only environmental problem faced by the petrochemical industry. There are many situations where it is desirable to remove components including organic pollutants (such as hydrocarbons) and heavy metal contaminants from produced water and water run-off before this water is released as effluent.

30 It is also desirable to remove such pollutants from liquids other than water and also from gases (e.g. air).

35 According to a first aspect of the present invention, there is provided a material comprising a base formed substantially of cellulose fibres onto which is adsorbed a composition comprising one or more

-2-

aliphatic carboxylic acids having hydrocarbon chains consisting of 8 to 20 carbon atoms.

In preferred embodiments, the hydrocarbon chains of the one or more carboxylic acids consist of 10 to 18 carbon atoms. Particularly effective carboxylic acids have been found to include stearic acid $\text{CH}_3(\text{CH}_2)_{16}\text{COOH}$ and palmitic acid $\text{CH}_3(\text{CH}_2)_{14}\text{COOH}$.

According to a second aspect of the present invention, there is provided a method of producing a material comprising a base formed substantially of cellulose fibres onto which is adsorbed a composition comprising one or more aliphatic carboxylic acids having hydrocarbon chains consisting of 8 to 20 carbon atoms, wherein one or more cellulosic materials are mixed together with a powdered formulation of the one or more carboxylic acids.

The material of the first aspect of the present invention may be formed by mixing together one or more cellulosic materials, for example virgin pulp and wood chips, together with the one or more carboxylic acids in powder form and, optionally, latex. The mixing is preferably undertaken in a hammer mill, in which heat and friction assist the process whereby the carboxylic acid becomes adsorbed onto the cellulose fibres. It is thought that the carboxylic acids are adsorbed onto the surface of the cellulose fibres by way of the carboxyl $-\text{COOH}$ functional group, either through hydrogen bonding or through the formation of cellulose esters containing an $-\text{O}-\text{CO}-\text{R}$ group formed with the hydroxyl $-\text{OH}$ groups on the cellulose rings. However the carboxylic acids are bonded to the cellulose fibres, the result is that the material of the first aspect of the present invention comprises cellulose fibres from which project hydrophobic hydrocarbon chains. When the material is applied to a mixture of water and hydrocarbon pollutants, the hydrophobic

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hydrocarbon tails of the carboxylic acid residues serve to attract the hydrocarbon pollutants to the material and to repel water, thereby providing the required separation. The material, incorporating the hydrocarbon pollutants, can then be gathered up and used as a fuelstock.

According to a third aspect of the present invention, there is provided a filter material comprising a matrix in which is dispersed a granular formulation of the material according to the first aspect of the present invention.

The matrix may be fabricated from a number of materials, including non-woven fibrous materials, open-cell foam materials or a cotton or viscose gauze. The unloaded matrix advantageously has a density not greater than 0.25gcm^{-3} , and preferably from 0.01 to 0.18gcm^{-3} . A particularly preferred matrix has a thickness of around 3mm and a density in the region of 0.1gcm^{-3} . The granular formulation of the material of the first aspect of the present invention may be incorporated into the matrix by bombardment across a pressure gradient as described in EP 0 504 214, the disclosure of which is hereby incorporated by reference into the present application. By incorporating the material of the first aspect of the present invention into a matrix to form a filter material, the available active surface area is increased so as to aid efficiency. Furthermore, dispersion of the material in the contaminated fluid is reduced because it is held within the matrix. In some embodiments, webs of the filter matrix are loaded to a density of around 1kgm^{-2} ; a density of $.925\text{kgm}^{-2}$ has been found to be particularly effective in certain circumstances. In other embodiments, a density of around 0.5kgm^{-2} has been found to be effective, particularly where the web of filter matrix has a thickness in the region of 3 or 4mm. Once

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CLAIMS:

1. A material comprising a base formed substantially of cellulose fibres onto which is adsorbed a composition comprising one or more aliphatic carboxylic acids having hydrocarbon chains consisting of 8 to 20 carbon atoms.

2. A material as claimed in claim 1, wherein the one or more aliphatic carboxylic acids have hydrocarbon chains consisting of 10 to 18 carbon atoms.

3. A material as claimed in claim 1 or 2, wherein the one or more carboxylic acids are selected from the group comprising stearic acid and palmitic acid.

4. A filter material comprising a matrix in which is dispersed a granular formulation of the material claimed in claim 1, 2 or 3.

5. A filter material as claimed in claim 4, wherein the matrix comprises a non-woven fibrous material.

6. A filter material as claimed in claim 4, wherein the matrix comprises an open-cell foam materials.

7. A filter material as claimed in claim 4, wherein the matrix comprises a cotton or viscose gauze.

8. A filter column comprising a hollow core upon which is mounted an alternating stack of filter plates and discs of the filter material as claimed in any of claims 4 to 7, wherein the filter plates are adapted to allow passage of fluid from a circumferential region of the filter column to the hollow core by way of the discs of filter material.

9. A filter cartridge comprising a hollow core around which is wrapped one or more layers of a filter material as claimed in any one of claims 4 to 7.

10. A filter pod comprising a casing internally divided into two chambers by a carrier which supports

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at least one filter cartridge as claimed in claim 9, the carrier and the at least one cartridge being arranged so that fluid can only pass from one chamber to the other by passing through both the hollow tubular core and the filter material of the at least one cartridge.

11. A method of producing a material comprising a base formed substantially of cellulose fibres onto which is adsorbed a composition comprising one or more aliphatic carboxylic acids having hydrocarbon chains consisting of 8 to 20 carbon atoms, wherein one or more cellulosic materials are mixed together with a powdered formulation of the one or more carboxylic acids.

12. A method according to claim 11, wherein the one or more cellulosic materials are selected from the group comprising wood chips and virgin pulp.

13. A method according to claim 11 or 12, wherein latex is added to the one or more cellulosic materials and the one or more carboxylic acids.

14. A method according to any one of claims 11 to 13, wherein mixing takes place in a hammer mill.

15. A method of cleaning a fluid by contacting the fluid with a material comprising a base formed substantially of cellulose fibres onto which is adsorbed a composition comprising one or more aliphatic carboxylic acids having hydrocarbon chains consisting of 8 to 20 carbon atoms, wherein one or more cellulosic materials are mixed together with a powdered formulation of the one or more carboxylic acids.

16. A method according to claim 15, wherein the fluid is air.

17. A method according to claim 15, wherein the fluid is water.

18. A filter cartridge comprising a container having a fluid input and a fluid output and including therebetween a quantity of the material of claim 1, 2 or 3.

PATENT COOPERATION TREATY

PCT

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INTERNATIONAL PRELIMINARY EXAMINATION REPORT

(PCT Article 36 and Rule 70)

Applicant's or agent's file reference HL54557/001/CTV	FOR FURTHER ACTION	See Notification of Transmittal of International Preliminary Examination Report (PCT/IPEA/416)
International application No. PCT/GB97/01319	International filing date (day/month/year) 14/05/1997	Priority date (day/month/year) 16/05/1996
International Patent Classification (IPC) or national classification and IPC B01D39/18		
Applicant AXHOLME RESOURCES LIMITED et al.		

<p>1. This international preliminary examination report has been prepared by this International Preliminary Examining Authority and is transmitted to the applicant according to Article 36.</p> <p>2. This REPORT consists of a total of 5 sheets, including this cover sheet.</p> <p><input checked="" type="checkbox"/> This report is also accompanied by ANNEXES, i.e., sheets of the description, claims and/or drawings which have been amended and are the basis for this report and/or sheets containing rectifications made before this Authority (see Rule 70.16 and Section 607 of the Administrative Instructions under the PCT).</p> <p>These annexes consist of a total of 5 sheets.</p>
<p>3. This report contains indications relating to the following items:</p> <ul style="list-style-type: none"> I <input checked="" type="checkbox"/> Basis of the report II <input type="checkbox"/> Priority III <input type="checkbox"/> Non-establishment of opinion with regard to novelty, inventive step and industrial applicability IV <input type="checkbox"/> Lack of unity of invention V <input checked="" type="checkbox"/> Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement VI <input type="checkbox"/> Certain documents cited VII <input type="checkbox"/> Certain defects in the international application VIII <input type="checkbox"/> Certain observations on the international application

Date of submission of the demand 05/11/1997	Date of completion of this report 05.08.98
Name and mailing address of the IPEA/ <div style="display: flex; align-items: center;"> <div> European Patent Office D-80298 Munich Tel. (+49-89) 2399-0, Tx: 523656 epmu d Fax: (+49-89) 2399-4465 </div> </div>	Authorized officer Persichini, C Telephone No. (+49-89) 2399-8617



INTERNATIONAL PRELIMINARY EXAMINATION REPORT

International application No. PCT/GB97/01319

I. Basis of the report

1. This report has been drawn on the basis of (*substitute sheets which have been furnished to the receiving Office in response to an invitation under Article 14 are referred to in this report as "originally filed" and are not annexed to the report since they do not contain amendments.*):

Description, pages:

4-20 as originally filed

1-3 as received on 20/07/1998 with letter of 17/07/1998

Claims, No.:

1-17 as received on 20/07/1998 with letter of 17/07/1998

Drawings, sheets:

1-9 as originally filed

2. The amendments have resulted in the cancellation of:

- ☐ the description, pages:
- ☐ the claims, Nos.:
- ☐ the drawings, sheets:

3. ☐ This report has been established as if (some of) the amendments had not been made, since they have been considered to go beyond the disclosure as filed (Rule 70.2(c)):

4. Additional observations, if necessary:

**INTERNATIONAL PRELIMINARY
EXAMINATION REPORT**

International application No. PCT/GB97/01319

V. Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement

1. Statement

Novelty (N)	Yes:	Claims	4-17
	No:	Claims	1-3
Inventive step (IS)	Yes:	Claims	
	No:	Claims	1-17
Industrial applicability (IA)	Yes:	Claims	1-17
	No:	Claims	

2. Citations and explanations

see separate sheet

1. DE-A-2 358 808 (D1) discloses a filter material (a material which is placed in a container and serves for filtering out contaminants from a fluid which is passed through the container is nothing else than a filter material; see D1, page 6, lines 9-13) comprising a matrix (Chambers English Dictionary: matrix = "that in which anything is embedded", eg a container) in which is dispersed a granular formulation of a material (according to eg claim 1 of D1 the modified cellulose mass is formed into small sized particles ["kleinteilig"]; it does not seem that there is a difference between "small sized particles" and a "granular formulation") comprising a base formed substantially of cellulose fibres onto which is adsorbed a composition comprising one or more aliphatic carboxylic acids having hydrocarbon chains consisting of 8 to 20 carbon atoms (see (D1), claim 1 and p.5, lines 1-5 and Example 16).

Claim 1 thus seems to lack novelty (Art.33(2) PCT).

However, even if a difference could be seen between the small sized particles of D1 and the "granular formulation" of claim 1, this difference would not be based on an inventive step under Art.33(3) PCT.

With regard to the handling and to the fluidic conditions (especially with regard to the pressure drop) the pelletization of filtering materials which are placed in a container and the filter activity of which is due to the adhesion of the components to be filtered (adsorption, absorption) to the surface of this material is usual. It is, therefore, evident for the skilled person to form pellets or granules from such a mass, if it has to be placed as a filtering fixed bed into a container.

2. The subject-matter of Independent method claim 10 differs from the teaching of (D1) on account only of the fact that the carboxylic acid is in powder form. In the absence, however, of any surprising effect (the advantages and disadvantages linked with the deposition of the carboxylic acid in dissolved form and in powder form, respectively, are well known in the art and consequently not surprising) attaching to this step, it does not represent an inventive advance over the process of (D1). Claim 11 thus lacks inventive step (Art.33(3) PCT).
Analogous arguments apply to the subject-matter of independent method (use) claim 15.

3. With regard to document (D1) and the general knowledge of the man skilled in the art, the dependent claims do not appear to contain any features which, in combi-

nation with the features of any claim to which they refer, meet the requirements of the PCT in respect of novelty or inventive step.